

**Elyria Site
NOx from Dried Precipitated Powders
Action Plan and Business Impact**



February 15, 2016

DATE

Background

- After the December 2015 production of Cu 6081 revealed that calcination can produce NO_x, other dried material calcined on the North calciners were evaluated
- Data has been obtained from Elyria, Beachwood and Iselin labs
- **All other products evaluated have potential to produce NO_x, but at significantly lower levels**
- The data conflicts with the conventional understanding of these processes
- ➔ ***Filtration and washing do not eliminate the potential for NO_x generation***

Potential Products Affected

All CuCr
Cu 5020 P / FT-BYD
X540T
Cu0811T
ML-1 Transfers (S1-81, BV0307, R3-11G)

These calciners are not currently permitted for NO_x and do not have equipment to control the potential NO_x emissions

Regulatory Requirements

- A source would be *de minimis* if it has the ***potential*** to emit less than ten pounds per day. A permit modification would not required but there must be supporting documentation to prove the daily potential emissions.
- A source can have a potential to emit of 10 ton/year for a criteria pollutant (which NO_x is) before control equipment must be installed. A permit modification would be required

The site currently does not have testing data to prove the potential daily NO_x emissions

Current Action Plan

- Production was stopped on the North calciners effective 2/12/16
- Parallel Actions:
 - Plan to run multiple products on rotary calciner #5 and have a third party test for NOx before entering the scrubbers. Target date week of March 1st
 - Working with a toller to qualify impacted products.
 - 6081 already qualified
 - Target end of March to qualify remaining products
 - Evaluating the capability of GCP/N Safety Engineering Wyandotte to quantify the NOx in the impacted products

Business Impact

- Cu-6081 – No impact. Toller will produce
- FT-BYD – 54 mt firm order to ship in July and other upsides that are not yet quantified. Scheduled to start early April.
- X-540 – 54 mt forecasted order for October.
- Impacts CCR additive production of 40k lb of 4010
- Trials for S1-81 and Cu-0811T would be delayed

Back Up



NO_x from Dried Precipitated Powders

Noemi Trent, Jack Pettry, Will Tuttle (Elyria G-CCP/OM)

Summary

- After AIMS case 0084-NOPS-15-0163 revealed Cu 6081 calcination can produce NO_x, other dried material calcined on the North calciners were evaluated
- Data has been obtained from Elyria, Beachwood and Iselin labs
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→ ***Filtration and washing do not eliminate the potential for NO_x generation***

Potential Products Affected

All CuCr
Cu 5020 P
Cu 0535 P
Cu 0808 P

ML-1 Transfers (S1-81, BV0307, R3-11G)

Cu 6081 P and Subsequent Investigation

- Cu 6081 was scaled up from R&D to Elyria with no expectation of NO_x
- Initial calcinations occurred without incident
- Upon calcination rate increase, visible NO_x was observed
- Analysis of calciner feed indicates material inherently contain insoluble nitrates in the 2-4% range
- Conventional understanding was that washing removed nitrate compounds
 - As a precaution, other current products were analyzed for insoluble nitrate levels
 - None were as high as Cu 6081, but all contained trace levels
 - ➔ *Whether these trace levels were capable of producing NO_x was not immediately clear*

Testing of Dried Powders

- Two techniques were used to analyze the powders:

Test	Site	Measures	Limitations
Kjeldahl	Elyria	solid nitrate content	Does not identify specific compound
TGA-MS	BW/ Iselin	identifies off gas during calcination	Doesn't quantify off gas concentration, lower detection limit unknown



**Decomposition Temperatures
T_d for Nitrate Compounds**

Compound	T _d [C]
Sodium Nitrate ¹	~740
Ammonium Nitrate ²	~260
Copper Nitrate ³	~250

Typical calcination temperatures: 350 – 550 C

- Kjeldahl: trace amounts of nitrates in all dried precipitated powders
 - However, not all nitrate compounds would form NO_x at process temperature
- TGA-MS: Inconsistent results, but very low levels of nitrates in powders did show trace NO_x during measurement.
 - Unclear how results translate to production process

Product	Typical wt% NO ₃ ⁻	NO _x detected by TGA-MS?
Cu 6081 P Dried	1 – 5	Yes, significant
Cu 0535 P Dried	0.2 – 1.5	Yes, trace
Cu 5020 P	0.1 – 0.6	Sometimes, but trace
Cu 0808 P Dried	0.1 – 0.6	Sometimes, but trace

Quantitative Measurement of Exhaust Stack Needed

- Improvised measurement: handheld area NOx meter held up to RC2 exhaust stack while Cu 0539 P (Cu/Mn/Al oxides) was running
 - > 20 ppm NO₂ measured (detection limit exceeded)
 - RC1 stack was too tall to measure, but after-filter seal was leaking ~2 ppm NO₂ . Cu 1885 P (CuCr) was running
 - RC1 exhaust directly measured while running empty, 0 ppm NO₂
 - NOx not inherent to process air
- Site management team informed
- More precise measurements are needed so that the concentration of NOx in the exhaust can be quantified.
- Recommendation: outside company needed for measurements